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Shelf life of powdered *Campomanesia adamantium* pulp in controlled environments

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ABSTRACT

The objective of this study was to evaluate the shelf life of powdered *guavira* pulp obtained by a foam mat drying process. The dehydrated *guavira* pulp was packed into low density polyethylene (LDPE) bags and stored under two controlled conditions: environmental (25 °C, RH 75%) and accelerated (35 °C, RH 90%) for 90 days. The shelf life was accompanied by carrying out the following analyses every 10 days: moisture content, water activity, vitamin C content, pH and titratable acidity. Vitamin C was the quality attribute used to determine the shelf life of the product, by determining its degradation kinetics as a function of storage time. The linear regression data showed that the vitamin C degradation reaction fitted the zero and first order kinetic models. The shelf life of the powdered guavira pulp under environmental conditions was approximately 49 days, and under accelerated conditions (35 °C) 45 days. The Q10 was equal to 1.09, predicting a shelf life similar to that found under environmental conditions. The moisture content for these conditions was 10.0% e 5.4% for 35 °C and 25 °C, respectively. The above demonstrate the efficiency of the accelerated test in predicting the shelf life of the product.

1. Introduction

Guavira (*Campomanesia adamantium*), also known as *gabiroba, guabiroba, guabiroba-do-campo* or *guariroba,* belongs to the Myrtaceae family and is of Brazilian origin growing in various regions of Brazil such as the savanna region (Porto & Gulias, 2010). The leaves of *C. adamantium* are used as infusion in the treatment of diarrhoea and bladder diseases (Cardoso et al., 2010). *Guavira* fruits have an agreeable flavour and aroma as well as elevated vitamin contents (Ramos, Cardoso, & Yamamoto, 2007) and are widely used in the production of homemade liqueurs, juices & sweets (Cardoso et al., 2010). However they are highly perishable and this fact together with a lack of post-harvest treatments are factors making its conservation difficult and contributing to its waste.

Of the food conservation processes mostly used, dehydration makes it possible to extend the shelf life, thus promoting the availability of a product for a more prolonged period; in addition it reduces the cost of packaging, transport and storage due to a reduction in weight and volume (Kadam et al., 2011). Amongst the dehydration methods, the foam mat drying process favours the drying of liquid or semi-liquid foods, which are transformed into stable foams by way of vigorous agitation and by incorporation of foaming agents for subsequent drying (Kadam, Wilson, & Kaur, 2010; Karin & Wai, 1999; Sankat & Castaigne, 2004).

According to Moura, Berbari, Germer, Almeida, & Fefim (2007), the shelf life of a food is defined by the time for which the product, stored under determined temperature conditions, presents alterations considered, up to a certain point, acceptable by the manufacturer, consumer and current food legislation.

Many products show prolonged shelf lives, making their experimental determination difficult. However, the existence of accelerated shelf life tests represents an alternative, and consists of storing the product to be studied under defined and controlled environmental conditions, so as to accelerate the rates of transformation (García-García, López-López, & Garrido-Fernández, 2008).

One way of evaluating the shelf life of a food is by establishing a quality index. For this purpose, the main quality parameters should be considered, as also the degree of deterioration necessary to establish the end of the shelf life (Sanjuán, Bom, Clemente, & Mulet, 2004). The shelf life depends on extrinsic factors such as processing, packaging properties, temperature and relative humidity of the environmental air, luminosity and headspace conditions, as well as intrinsic factors of the food such as acidity, available oxygen, additives, level of microbial contamination, redox potential and water activity (Escobedo-Avellaneda, Velazquez, Torres, & Welti-Chanes, 2012).

Some of the main parameters considered in predicting shelf life are colour, ascorbic acid content, moisture content and pH value (Arlindo, Queiroz, & Figueiredo, 2007; Galdino, Queiroz, Figueiredo, & Silva, 2003; Gomes, Figuerêdo, & Queiroz, 2004).

Thus the objective of the present study was to evaluate the shelf life of powdered *guavira* pulp produced by a foam mat process,

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employing accelerated tests as a function of the ascorbic acid content.

2. Materials and methods

Guavira fruits were acquired in the town of Bela Vista, MS, Brazil (Latitude −22° 06′ 32″ and Longitude −56° 31′ 16″) and transported to the Food Technology Laboratory of the Faculty of Engineering/UFGD, Brazil. The fruits were selected according to their degree of ripeness and physical integrity, washed and sanitized with 0.66% sodium dichloroisocyanurate dehydrate (Sumaveg). After sanitization the fruits were immersed in water at 70 °C for 5 min, drained, manually crushed and the pulp separated from the seeds and skin. The pulp was then packaged in rigid polypropylene containers and stored at −22 °C until use.

2.1. Sample preparation

Guavira foam was produced by mixing 100 g pulp with 1% citric pectin, 2% Emustab (product based on distilled monoglycerides, sorbitan monostearate and polysorbate 60) and 1% Super Liga neutral (product based on sucrose, carboxymethylcellulose and guar gum) and agitated at 1,050 rpm for 20 min in a mixer (Black & Decker Power Pro) at room temperature. The foam was transferred to stainless steel trays so as to form 2.00 mm thick layers and placed in the dryer (NG científica) at 74 °C, with hot air circulation at a velocity of 0.5 m/s for 120 min.

The dehydrated foam was ground in an industrial blender (Skymsen) to form a powder.

2.2. Shelf life study

For the shelf life study, 25 g samples of powdered *guavira* pulp were packed into 120 × 120 mm (10 μm thick) low density polyethylene (LDPE) bags. The study was carried out under two controlled environmental conditions: (1) relative humidity of 75% and temperature of 25 °C (environmental conditions) and (2) relative humidity of 90% and temperature of 35 °C (accelerated conditions).

The environmental humidity conditions (relative humidity) were reproduced in desiccators containing saturated solutions of sodium chloride ($a_w = 0.75$) for the environmental conditions (1) and barium chloride ($a_w = 0.90$) for the accelerated conditions (2). The *guavira* powder packages were distributed in the desiccators so that they did not obstruct the circulation of the moist air inside the systems, avoiding direct contact with the saturated solutions. The temperature conditions were maintained constant by placing the desiccators inside BOD (biochemical oxygen demand) chambers. The storage period was 90 days and during this period, three packages of samples were removed every 10 days for evaluation of the moisture content, water activity, vitamin C content, pH value and titratable acidity. The analyses carried out at zero time were considered to be the standard condition.

2.3. Physical and chemical analyses

The moisture content was determined using a gravimetric method in an incubator with air circulation according to the AOAC method 15010 (1975), adapted for 70 °C and 24 h to avoid sample caramelization; the following parameters were measured, water activity (a_w) by direct measurement in a hygrometer (Aqualab, Decagon, series 3.0); vitamin C content by Tillmans method with a solution of 2,6-dichlorophenolindophenol, according to AOAC method 967.21 (2000); pH by direct reading on a digital pH-meter (Labmeter) and titratable acidity by AOAC method 942.15 (1997).

2.4. Determination of the reaction order

To determine the reaction order and its velocity constant, the values obtained for the % vitamin C degradation were plotted as a function of storage time, and linear regression was carried out corresponding to the values for k (reaction velocity) for each temperature and each reaction order (Eqs. (1) and (2)).

$$\frac{dA}{dt} = k_0 \tag{1}$$

$$\frac{dA}{dt} = k_1 A \tag{2}$$

In the integrated form and rearranged in the form of the equation of the curve, one obtains (Eqs. (3) and (4)):

$$A = -kt + A_0 \tag{3}$$

$$\ln A = -kt + \ln A_o \tag{4}$$

Eq. (5) was used to determine Q_{10} and Eq. (6) for the shelf life estimate.

$$Q_{10} = \frac{k(T + 10^\circ C)}{k(T)} = \frac{t_f(T)}{t_f(T + 10^\circ C)} \tag{5}$$

$$t_f = \frac{\ln(A_o/A_f)}{k} \tag{6}$$

where Q_{10} is the quotient between the reaction velocity at a determined temperature and a temperature 10 °C higher; k is the reaction velocity, T (°C) is the temperature, t_f the shelf life, A_0 the ascorbic acid content at the start of storage (653.00 mg/100 g) and A_f the ascorbic acid content after 55% degradation during storage (Labuza & Schmidl, 1985).

2.5. Statistical analysis

The results obtained were statistically evaluated using the variance analysis (ANOVA) and the means compared by Tukey's test using version 7.0 of the Statistica program.

3. Results and discussion

3.1. Moisture content

Fig. 1 shows the results obtained for moisture content. It can be seen that there was greater water absorption in the accelerated state (35 °C and 90% RH), increasing by about 4.7 times as compared to the product at zero time, whereas under environmental conditions, this increase was about 2.5 times. According to Arlindo et al. (2007), the hygroscopic characteristics of some foods depend mainly on their chemical composition and storage conditions (air relative humidity), which explains the greater increase in moisture content under the accelerated conditions. Few papers can be found in the literature concerning the shelf life of dehydrated powdered products stored in controlled environments. Thus, the increase in moisture content of the product at the end of the experiment can be attributed to the permeability of the packaging materials, favouring the absorption of moisture from the environment of the controlled storage system.

Powdery products have presented, in general, low moisture contents, in the 4–6% interval. In this study, in the accelerated condition (35 °C) the product showed 6% of moisture content in 26 days of storage; under these conditions, the vitamin C retention was 75% of initial value. In environmental conditions (25 °C), the same moisture content was obtained in approximately 49 days and the product showed 45% vitamin C retention (Figs. 1 and 3).

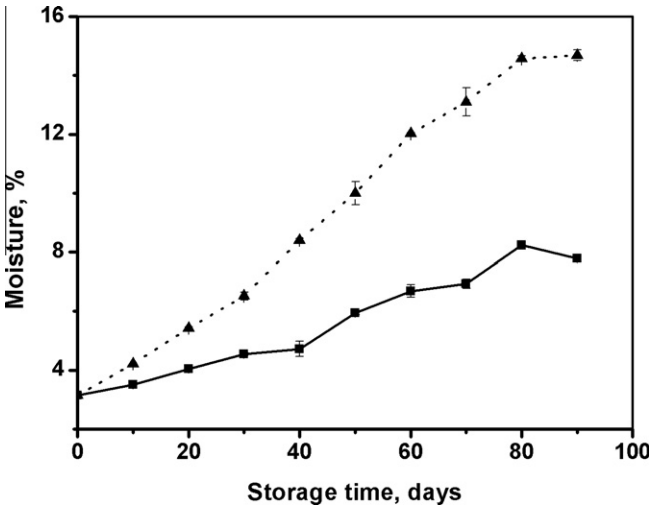

Fig. 1. Mean values for the moisture content (%) of the samples of powdered *guavira* pulp under environmental (■) and accelerated (▲) conditions for 90 days.

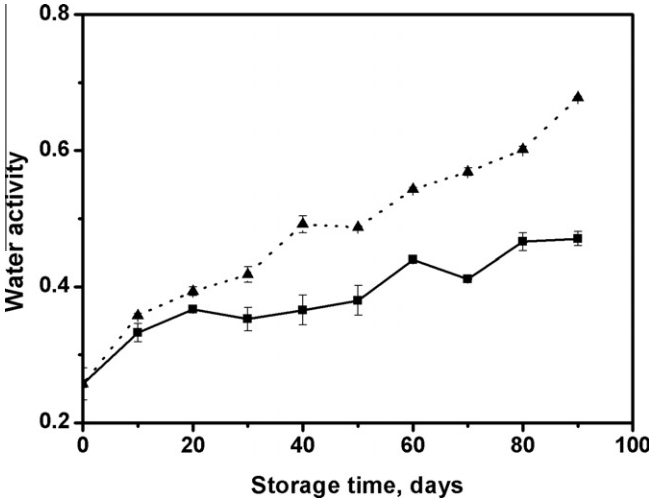

Fig. 2. Mean values for the water activity (adm) of the samples of powdered *guavira* pulp under environmental (■) and accelerated (▲) conditions for 90 days.

3.2. Water activity

Fig. 2 shows the results obtained for water activity of the powdered *guavira* pulp, indicating that, as for moisture content, the water activity increased during storage, being greater under accelerated conditions (0.680) than under environmental conditions (0.470).

This difference can be attributed to the relative humidity of the air in the storage environment. According to Garcia, Padula, and Sarantopulos (1989) as cited by Gomes et al. (2004), the type of packaging material used for food products constitutes a barrier that impedes or hinders contact between the food and the external environment. Nevertheless the permeability of the packaging materials should be considered. Depending on the permeability rates of water vapour and oxygen, greater absorption of moisture can occur from the environment, consequently influencing the water activity and justifying the greater absorption of water under accelerated conditions.

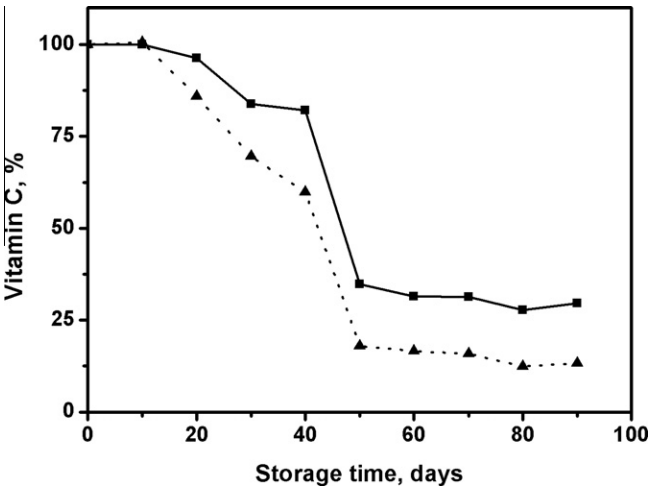

Fig. 3. Vitamin C degradation as a function of storage time – Environmental conditions: 25 °C and 75% RH (■) and accelerated conditions: 35 °C and 90% RH (▲).

According to Khalloufi, Giasson, and Ratti (2000), water activity constitutes an important concept in the food industry, since it is related to the microbiological and physicochemical stability of the deteriorative reactions, whereas Esse and Saari (2004) explain that for a low water activity product, the degradation of vitamins, such as ascorbic acid, is reduced.

3.3. Ascorbic acid

According to Taoukis and Labuza (1996), vitamin losses, pigment oxidation and microbial growth all follow a first order pattern, where the rate of quality loss is directly related to the remaining quality. In the present study the ascorbic acid (vitamin C) stability was studied due to its importance in the human diet. In addition, since it is considered to be the most chemically unstable vitamin, one can consider that if the ascorbic acid is retained in the food, the other nutrients will also be retained. Thus its retention is considered to be an index of nutritional quality maintenance during food processing and storage (Hiatt, Taylor, & Mauer, 2010). In this work, the vitamin C content obtained at zero time was considered as 100% for the initial (A_0) condition and 45% for the final condition (A_f). The final condition was defined considering that 15.0 g powder reconstituted in 200 ml water at the start of storage provided approximately 98 mg ascorbic acid. Since the recommended daily allowance for adults is 45 mg, it was considered that the product with 45% of vitamin C retention would still provide the recommended daily vitamin C allowance.

Fig. 3 shows that the ascorbic acid content of the powdered *guavira* pulp decreased sharply between the 10th and 50th days of storage under accelerated conditions, and between the 20th and 50th days of storage under environmental conditions, and then remained practically constant up to the end of storage, presenting first order degradation kinetics up to the 50th day of storage and then zero order kinetics up to the end of storage under both storage conditions. Although significant vitamin C degradation is represented by the first order kinetics, the overall degradation velocity of the system was calculated to check whether or not the influence of zero order kinetics.

For variable order reactions (Levenspiel, 1974), the overall degradation velocity of CA may be calculated by the sum of the individual velocities (Eq. (7)). Therefore, we applied zero order equations (Eq. (1)) and first order (Eq. (2)) separately, obtaining the velocity constants k_0 and k_1.

Table 1
Kinetic parameters for vitamin C degradation and shelf life.

T (°C)	k_0 (day^{-1})	$R^2_{k_0}$	k_1 (day^{-1})	$R^2_{k_1}$	A_0 (%)	A_f (%)	Q_{10}	t_{f_1} (days)	t_{f_g} (days)
25	0.0037	0.836	0.0163	0.661	100	45	1.09	48.99	48.82
35	0.0091	0.968	0.0177	0.996	100	45		45.11	44.76

K_0 = zero order reaction constant. $R^2_{k_0}$ = correlation coefficient for zero order kinetic. K_1 = first order reaction constant. $R^2_{k_1}$ = correlation coefficient for first order kinetic. A_0 and A_f = vitamin C content at zero time and after losing 55%, respectively. Q_{10} = shelf life quotient. t_f = shelf life for first order equation and t_{fg} = shelf life for global equation.

Table 2
Values for the pH and titratable acidity of powdered *guavira* pulp during storage under environmental (25 °C and 75% RH) and accelerated (35 °C and 90% RH) conditions.

Time (days)	pH		Titratable acidity (%)	
	Environmental	Accelerated	Environmental	Accelerated
0	4.78 ± 0.23^{a}	4.78 ± 0.23^{a}	15.14 ± 0.50^{a,h}	15.14 ± 0.50^{a}
10	4.34 ± 0.01	4.29 ± 0.01	16.13 ± 0.07^{g,h}	16.16 ± 0.46^{a}
20	4.47 ± 0.01^{b}	4.33 ± 0.01^{b}	15.24 ± 0.21^{h}	15.58 ± 0.62^{a}
30	4.39 ± 0.01	4.34 ± 0.01^{b}	18.92 ± 0.80^{b,a,g,h}	17.41 ± 0.21^{a,f}
40	4.31 ± 0.04	4.14 ± 0.01^{c}	17.29 ± 0.38^{h}	17.22 ± 0.21^{a,f}
50	4.34 ± 0.02	4.23 ± 0.01	18.87 ± 0.89^{c,a,h}	15.74 ± 1.09^{a}
60	4.33 ± 0.03	4.12 ± 0.01^{c}	18.97 ± 1.56^{d,a,h}	23.06 ± 1.20^{c}
70	4.40 ± 0.01^{d}	4.24 ± 0.03	22.17 ± 0.27^{e}	21.21 ± 1.50^{c,b}
80	4.17 ± 0.01^{c}	3.93 ± 0.01^{d}	17.23 ± 1.33^{h}	19.32 ± 1.12^{a,f,b}
90	4.17 ± 0.01^{c}	3.94 ± 0.02^{d}	21.33 ± 1.06^{f,g}	19.39 ± 1.06^{e,f,b}

Different letters in the same column indicate significant difference according to Tukey's test at the 5% level.

$$\left(\frac{dA}{dt}\right) = k_0 + k_1 A \tag{7}$$

In the integrated form, one obtains:

$$-\ln\left(\frac{k_0 + k_1 A_0}{k_0 + k_1 A}\right) = k_1 t \tag{8}$$

The results obtained from the first order degradation velocity for the shelf life of the product with 45% retention of vitamin C (28.99 days) did not differ significantly ($p > 0.05$) from those obtained from the overall degradation velocity equation (48.82 days) and experimental data (approximately 48 days). This shows that the first order kinetics prevails in the vitamin C degradation. However, the shelf life prediction from the reaction velocity equations (Eqs. (1) and (2)) reproduces only experimental values close to 50% degradation.

According to Hiatt et al. (2010), the literature has reported zero order, first order and second order kinetic models for the vitamin C degradation, however, these models are commonly related to high a_w foods or ascorbic acid solutions. However, these models may not be applicable to powder systems which have moisture absorption during storage. In this work, the reaction fitted the first order kinetic model up to the 50th day, and then zero order up to the end of the experiment (90 days). For the prediction of the product shelf life of the obtained values for vitamin C degradation between zero and 50 days were considered; thereafter, the vitamin C degradation was considered negligible in relation to time. First order kinetic behaviour is frequently observed for vitamin degradation, whereas zero order kinetic behaviour is observed when the diffusion of certain participants of the reaction is limited (Taoukis & Labuza, 1996). According to Nagy (1980), after consumption of the free oxygen in the packages, anaerobic reactions become predominant, including that of ascorbic acid degradation, but at a reduced velocity as compared to that occurring under aerobic conditions, which can explain the reduction in the oxidation reaction in the end of storage. Under these conditions, the ascorbic acid decomposes into 2,5-dihydro-2-furanoic acid, which degrades to carbon dioxide and furfural. For its part furfural undergoes polymerisation as an active aldehyde, and can combine with amino acids, influencing product browning (Shaw, Nagy, & Rouseff, 1993; Solomon, Svanberg, & Sahlström, 1995).

Table 1 shows the ascorbic acid degradation kinetics of powdered *guavira* pulp. The values for the constant (k) indicate that the reaction velocity increases with increase in temperature. At 35 °C the storage time was 45 days, which, multiplied by the factor of 1.09 given by Q_{10}, resulted in a shelf life of 49 days under storage conditions at 25 °C. The moisture content for these conditions was 10.0% and 5.4% for 35 °C and 25 °C, respectively.

According to Silva, Gurjão, Almeida, Bruno, & Pereira, 2008, the oxidation of ascorbic acid is mainly influenced by an increase in temperature, whereas Lee and Kader (2000) reported that this vitamin was easily oxidised in aqueous media and in the presence of oxygen, metal ions and alkaline pH values, amongst other factors. Galdino et al. (2003) explained that this behaviour could be attributed to the low protection provided by polyethylene, making the material susceptible to the effects of micro-environments created in the setting up of trials, allowing for the migration of moisture from the environment until reaching equilibrium.

3.4. pH and acidity

Table 2 shows the mean values obtained for the pH and titratable acidity of the powdered *guavira* pulp stored in polyethylene packages. A decrease in the pH value with time can be seen under both storage conditions, reaching values of 4.17 and 3.94 at the end of the storage period. According to Martins, Jongen, and Van Boekel (2000), non-enzymatic browning reactions are favoured by high pH values, and are inhibited at pH values below 5.0. The influence of pH was also observed with respect to enzymatic browning. Polyphenoloxidase is the main browning enzyme present in the majority of vegetable matter, and requires pH values between 5.0 and 7.0 for its activity. It can be irreversibly inactivated at pH values below 3.0 (Martinez & Whitaker, 1995). In addition, the pH also affects the stability of vitamin C. According to Lipasek, Taylor, and Mauer (2011), in more basic conditions, the lactone rings structure of the vitamin degrades more quickly.

Acerola pulp dehydrated in a spouting bed and stored in polyethylene packages at room temperature for 60 days showed no difference in the pH value between the initial and final samples (Gomes et al., 2004).

With respect to acidity (Table 2), there was no significant ($P > 0.05$) difference up to the 60th day of storage under environmental condition and the 50th under accelerated condition. However, as from the 70th and 60th day under environmental and accelerated conditions, respectively, the samples showed oscillations. This variation could be attributed to the buffering effect of the samples, since according to Chitarra and Chitarra (2005), this capacity allows for considerable variations in acidity without presenting measurable pH variations.

4. Conclusions

The absorption of water vapour by dehydrated *guavira* pulp stored in low density polyethylene bags, was proportional to the increases in temperature and relative humidity of the air. The shelf

life study of the powdered *guavira* pulp as a function of ascorbic acid dehydration showed first and zero order reactions. The shelf life of the powdered *guavira* pulp stored in LDPE bags under environmental conditions with 45% reduction in vitamin C was 49 days, and 45 days under accelerated conditions (35 °C), with a Q_{10} of 1.09, which predicts a shelf life of 49.09 days under normal storage conditions.

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